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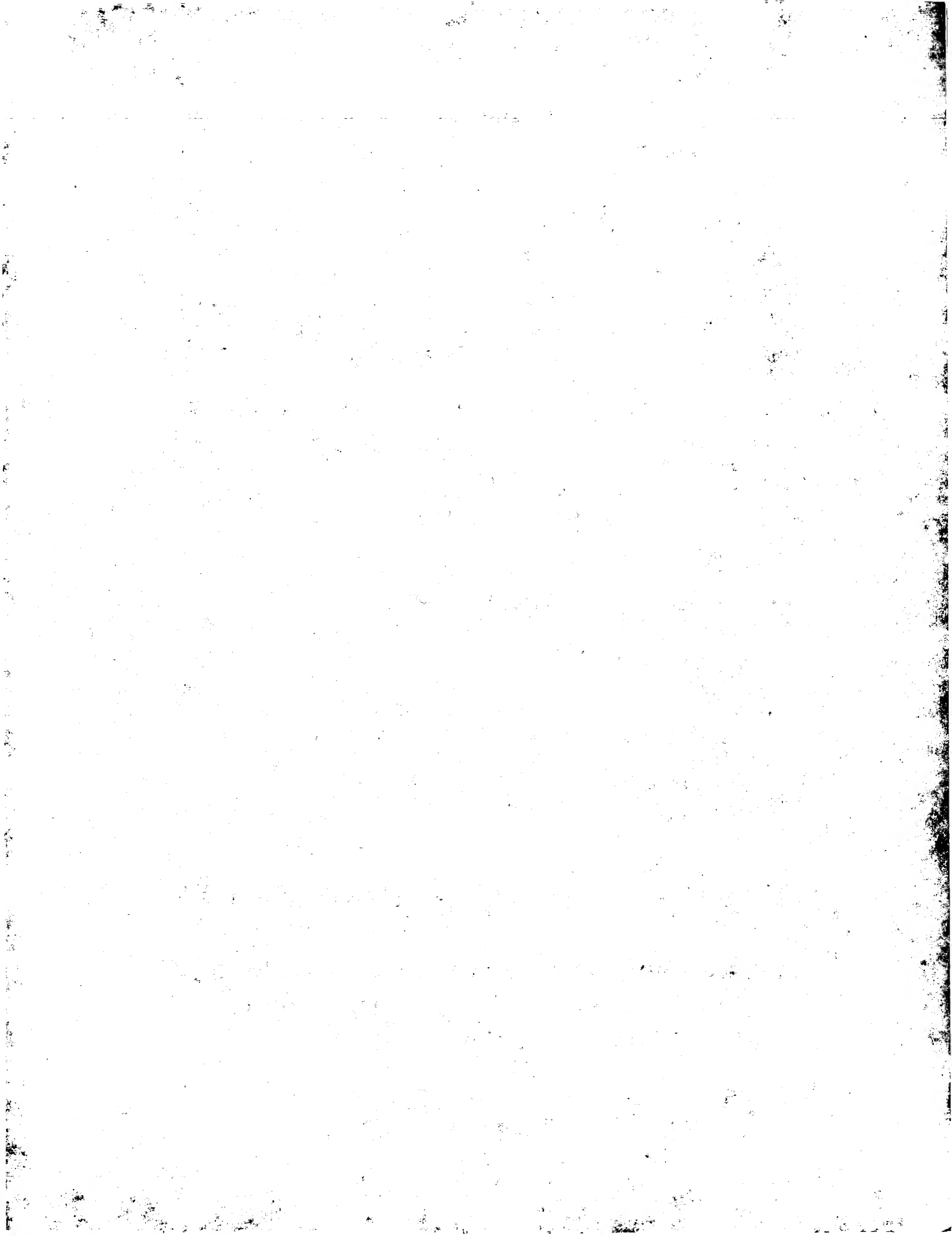
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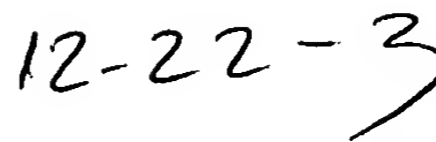
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Express Mail No. <b>EV 346 810 232US</b>		
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4717-6600	10/664,782	<b>Submission of Certified Priority Document EP 02292518.4</b>

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Express Mail No. **EV 346 810 232US**

**IN THE UNITED STATES PATENT AND TRADEMARK OFFICE**

In re application of: Christophe MALEVILLE et al.

Confirmation No. 4834

Application No: 10/664,782

Group Art Unit: 2855

Filing Date: September 16, 2003

Examiner:

For: METHOD FOR EVALUATING PARTICLE  
CONCENTRATIONS IN A CLEAN ROOM OR  
MACHINE MINI-ENVIRONMENT

Atty. Docket No.: 4717-6600

**SUBMISSION OF CERTIFIED PRIORITY DOCUMENT**

Commissioner for Patents  
P.O. Box 1450  
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Sir:

Applicants have claimed priority under 35 U.S.C. § 119 of European Application No. 02292518.4 filed October 11, 2002 in Europe. In support of this claim, a certified copy of said application is submitted herewith.

No fee or certification is believed to be due for this submission. Should any fees be required, however, please charge such fees to Winston & Strawn LLP Deposit Account No. 50-1814.

Respectfully submitted,

Date: 12/18/03

Allan A. Fanucci (Reg. No. 30,256)

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Die angehefteten Unterlagen stimmen mit der ursprünglich eingereichten Fassung der auf dem nächsten Blatt bezeichneten europäischen Patentanmeldung überein.

The attached documents are exact copies of the European patent application described on the following page, as originally filed.

Les documents fixés à cette attestation sont conformes à la version initialement déposée de la demande de brevet européen spécifiée à la page suivante.

**Patentanmeldung Nr.    Patent application No.    Demande de brevet n°**

02292518.4

Der Präsident des Europäischen Patentamts;  
Im Auftrag

For the President of the European Patent Office

Le Président de l'Office européen des brevets  
p.o.

**R C van Dijk**





Anmeldung Nr:  
Application no.: 02292518.4  
Demande no:

Anmeldetag:  
Date of filing: 11.10.02  
Date de dépôt:

Anmelder/Applicant(s)/Demandeur(s):

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Bezeichnung der Erfindung/Title of the invention/Titre de l'invention:  
(Falls die Bezeichnung der Erfindung nicht angegeben ist, siehe Beschreibung.  
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A method for evaluating a particle concentration in an atmosphere of a clean-room  
or a mini-environment

In Anspruch genommene Priorität(en) / Priority(ies) claimed /Priorité(s)  
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Staat/Tag/Aktenzeichen/State/Date/File no./Pays/Date/Numéro de dépôt:

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## **A Method for Evaluating a Particle Concentration in an Atmosphere of a Clean-Room or a Mini-Environment**

The present invention relates to a method for evaluating a particle concentration, such as a surface contamination or a atomic contamination especially of boron or phosphor, in an atmosphere of a clean room or a machine mini-environment.

A clean room is a work area with controlled temperature, humidity and particle concentration to protect sensitive equipment or products from contamination. Many different industries such as medical facilities or integrated circuit manufacturers require the use of clean rooms with a high quality.

Therefore, the clean rooms are equipped with various very expensive and complicated air supply cleaning systems providing the clean rooms with dust-free filtered air.

Nevertheless, due to a leakage in the system and due to the consistence of the air filters, a certain amount of boron and phosphor remains in or is added to the clean room atmosphere which can lead to a contamination of wafers. For instance, the contamination of boron or phosphor on a surface of a processed silicon wafer can amount to  $10^{12}$  atoms per  $\text{cm}^2$ . These particles can diffuse during thermal treatment into the silicon wafer surface leading to a change of the dopant concentration in this region affecting the characteristic of the processed chips. Therefore, it is necessary to know the particle concentration of the clean room.

In a conventional technology for monitoring boron or phosphor in a clean room environment, air of the clean room is lead through a liquid wherein a part of the boron or phosphor of the formed air bubbles dissolves in the liquid. After a relatively long period of time, e.g. a day, the liquid has picked up an amount of boron or phosphor which is high enough for analysing. Then the boron or phosphor content of the liquid is evaluated by a spectroscopy based analysis such as Atomic Spectroscopy or ICPMS.

This method reflects only in a relatively imprecise way the real boron or phosphor concentration of the air, because only an indefinite part of the actual boron or phosphor

content is solved in the liquid since most of the air including boron or phosphor escapes with the bubbles from the liquid before dissolving. It is a further disadvantage that this method has to be applied over a relatively long period of time in which only a mean value of the boron or phosphor of the clean room can be evaluated.

It is the object of the present invention to provide a method for evaluating a particle concentration in an atmosphere in the clean room reflecting a more real and precise particle concentration of the clean room in a short time.

This object is solved by a method for evaluating a particle concentration in an atmosphere of a clean room comprising the steps of exposing a surface of a test substrate, especially of a silicon wafer to said atmosphere, for a test time; catching and conserving a particle amount at the test substrate at the end of said test time; analysing the particle amount and comparing the analysed particle amount with the particle amount analysed on a reference substrate.

By this method, a certain particle amount on the surface of the test substrate closely corresponding to the particle concentration in the atmosphere of the clean room can be kept and fixed at the test substrate after a definable test time for analysing. This particle amount can show the state of the test substrate of a certain time of contamination of the test substrate. Then, a comparison between the analysed particle amount on the test wafer with that on the reference substrate provides a relative particle concentration in the atmosphere of the clean room in the pre-defined time period. This allows a conclusive determination of the contamination of the atmosphere.

The inventive method can be further used to monitor the particle concentration in the special mini-environment of a machine, such as a boron or phosphor contamination of the test substrate induced for instance by filters in the machine mini-environment.

In an embodiment of the invention, the method further comprises cleaning of the test substrate before exposing the test substrate to said atmosphere. In this cleaning step, a particle concentration on the surface of the test substrate can be removed resulting in a nearly particle free test surface at the time  $t=0$  at the beginning of an exposure of the test substrate to the atmosphere. So, the analysed particle amount directly corresponds to the

particle concentration in the atmosphere reflecting the period from the beginning to the end of the exposure of the test substrate of the atmosphere.

In a further embodiment of the invention, the step of catching the particle amount comprises covering the test surface of the test substrate with a surface of another substrate after the test time. The other substrate holds the particles which lay on, are absorbed or are migrated into a surface near region of the test substrate, and catches the particles in their actual position after the test time, making it possible to analyse exactly this amount to conclude from this amount to the particle concentration in the atmosphere of the clean room. The other covering substrate can be of another material than the test substrate.

In an advantageous example of the invention, said other substrate is a further test substrate, especially a silicon wafer, wherein said substrates form a test pair of substrates. Covering of the test substrate with an equal or a similar substrate has the advantage that the substrates have the same or a comparable particle catching behaviour. Furthermore, these substrates can easily be brought together to form, for instance, a bonded pair of substrates.

According to an advantageous variant of the invention, the step of conserving the particle amount comprises an annealing of the test substrate after catching the particle amount. By this annealing step, the caught particles can diffuse into the test substrate where they are conserved or fixed for a further analysing of the test substrate.

In a variant of the invention, said annealing is performed in a time range between about 1 to 3 hours, preferably in about 2 hours. During this time, the particles can diffuse into the test substrate creating a certain particle distribution in the test substrate which can be analysed to evaluate the caught particle amount. The annealing time of 2 hours allows a particularly favourable penetration depth of the particles.

In a further embodiment of the invention, said annealing is performed at a temperature between 800°C and 1050°C, preferably at 950°C. The diffusion activity of the particles is at this temperature high enough to get a good particle distribution in the test substrate which can be easily analysed. Especially at 950°C, a good relationship between the created particle distribution and the required energy for diffusion is achieved. The upper limitation of

temperature at 1050°C prevents a too homogenous distribution of the particles in the substrate and/or a too strong bonding of the substrates.

According to a specific embodiment of the invention, a test pair of substrates bonded to each other at the end of said test time is subsequently separated for analysing. In this way, the test surface which was exposed to the atmosphere for the test time and which state was conserved by a bonding step is made accessible for analysing the particle amount which was conserved at the substrate at the end of the test time.

In a further example of the invention, the method further comprises an abrasion of at least one of the back sides of a test pair of substrates bonded to each other. The abrasion of material from a back side of the test pair of substrates allows a stepwise backwards approach to the test surface which is conserved by bonding of the substrates to analyse the particle amount at least at the surface.

According to a further embodiment of the invention, analysing the particle amount comprises evaluating the atomic concentration profile of the test substrate. The atomic concentration profile gives a direct indication of a particle quantity included in and on the test substrate.

In a further advantageous embodiment of the invention, the atomic concentration profile is evaluated by a Secondary Ion Mass Spectroscopy. The Secondary Ion Mass Spectroscopy is a very precise and efficient analysing method for providing the atomic concentration profile of the test substrate.

In a further embodiment of the invention, the atomic concentration profile is evaluated at the test surface of the test substrate. Because the test surface is exposed to the atmosphere of the clean room its atomic concentration profile reflects well the particle concentration in the atmosphere.

In another preferable embodiment of the invention, the test surface is analysed over a thickness of about 100 to 500 nanometers, preferably 200 nanometers. This thickness gives adequate information about the particle amount in the test substrate. Especially, the

analysis thickness of 200nm allows an effective relationship between the expenditure of energy for analysing and the evaluated particle amount over this thickness.

According to another preferable variant of the invention, a series of test substrates is produced in timely intervals, preferably about every 30 minutes. This offers the possibility to monitor the change of the particle concentration in the atmosphere of the clean room or the mini-environment in the given intervals. In particular, the time intervals of every 30 minutes are highly expressive to get an overview of the particle concentration in the atmosphere of the clean room or the mini-environment over this period of time. So it is possible to evaluate a contamination rate in the clean room or the mini-environment.

In yet another preferable embodiment of the invention, the method further comprises cleaning of the reference substrate, preferably together with the test substrate. The cleaned reference substrate represents the state without particles on the substrate at the time  $t=0$  which can easily be compared with the test substrate. When the reference substrate is cleaned together with the test substrate, both substrates have the same starting conditions on their surfaces which makes the results of the following comparison of the substrates more precise.

In a further embodiment of the invention, the method further comprises covering of a reference surface of the reference substrate with a reference surface of another reference substrate. So, the initial or original condition on the reference surface of the reference substrate can be kept for analysing and getting reference values of an initial or original particle amount which can be compared with the particle amount of other test substrates.

According to another preferable embodiment of the invention, the method further comprises an annealing of the reference substrate. In this annealing step, an eventual particle amount on the reference substrate can diffuse into the substrate to conserve this particle amount in a region near the surface of the reference substrate.

In another advantageous variant of the invention, the annealing temperature and the annealing time of the annealing of the reference substrate are equal to an annealing temperature and an annealing time of the test substrate. By using the same annealing temperature and annealing time on the test substrate and on the reference substrate, the

measurement results on the substrates can be better compared after analysing of the substrates.

In yet another further advantageous embodiment of the invention, the method further comprises analysing a particle amount of the reference substrate. This particle amount can be compared with the evaluated particle amount of the test substrate.

In another embodiment of the invention, analysing the particle amount comprises evaluating the atomic concentration profile of the reference substrate. This atomic concentration profile can be compared with the test substrate.

In an advantageous variant of the invention, the atomic concentration profile of the reference substrate is evaluated by a Secondary Ion Mass Spectroscopy. This spectroscopy enables a precise evaluation of the particle amount in and on the reference substrate.

In a favourable embodiment of the invention, the atomic concentration profile of the reference substrate is evaluated at a reference surface of the reference substrate. This surface represents the initial or original particle amount on the reference substrate, which is a good indication of the particle amount on the reference substrate at the time  $t=0$ , which can be compared with the particle amount analysed on the test substrate.

According to yet a further embodiment, the reference surface of the reference substrate is analysed over a thickness of about 100 to 500 nanometers, preferably of about 200 nanometers.

This thickness corresponds to a preferable thickness for analysing of the test substrate and is therefore well suited for a further comparison between the reference substrate and the test substrate. Especially the thickness of 200 nm allows analysing of the particle amount in a relevant surface near part of the substrate.

An embodiment of the present invention will be described in the following with reference to the accompanying figures, in which

- Figure 1 shows steps of an embodiment of the method for evaluating a boron or a phosphor concentration in an atmosphere of a clean room or a machine mini-environment according to the present invention;
- Figure 2 shows schematically a concentration profile of a bonded test wafer pair; and
- Figure 3 shows schematically SIMS-concentration profiles of a test wafer and a reference wafer.

Figure 1 shows steps of an embodiment of the method for evaluating a boron or a phosphor concentration in an atmosphere of a clean room or a machine mini-environment according to the present invention. In the method silicon wafers 1, 3, 5 and 6 are handled. For a boron analysis n-type-silicon and for a phosphor analysis p-type-silicon is used. The substrate level  $N_s$  which is the bulk dopant concentration of the silicon wafers is lower than the level of the species which are to analyse.

In step a) of Fig. 1, two or more silicon wafers 1,3 which are shown standing erect or inclined in a wafer holder 10 are dived in a bath 8 containing a cleaning solution 9. The cleaning solution 9 removes inorganic and organic contamination on surfaces 2,4 of the silicon wafers 1,3. As cleaning solution 9, typical RCA cleaning solutions are used which can be complimented or replaced by other suitable solutions or mixtures of gases and solutions which are able to remove any contamination on the surfaces 2,4 of the silicon wafers 1,3. The cleaning solution 9 has a temperature between room temperature of about 19°C to 25°C and about 85°C and can be stirred by a stirrer (not shown) or recirculated to get a homogenous concentration of the components of the cleaning solution.

According to step b) of Fig. 1, reference wafers 3 and 6 are bonded by bringing their cleaned surfaces together immediately after cleaning of the wafers 3,6, and by covering of the surfaces with each other, whereas in step b') of Fig. 1, one of the cleaned silicon test wafers 1 and a further silicon test wafer 5 (not shown) are exposed to an atmosphere 7 of a clean room. The atmosphere 7 contains air which is contaminated with boron or phosphor. A part of the boron or phosphor atoms are adsorbed or deposited on the surface 2 of the silicon wafer 1.

In step c) of Fig. 1, a bonded test wafer pair is formed between the test wafers 1 and 5 by covering the test surface 2 by the other test surface 16 and bonding the test surfaces 2 and 16 of these substrates together. The reference wafer pair 3,6 keeps by this bonding step the initial clean condition of the surface 4 as a reference at its bonding interface, whereas the test wafer pair 1,5 has caught the boron or phosphor contaminants which are deposited on the test surfaces 2,16 of the test wafers 1,5 at the bonding interface of the test wafer pair.

In step d) of Fig. 1, the bonded wafer pairs 3,6 and 1,5 are annealed in an annealing equipment 11 at about 950°C for approximately two hours. In this annealing step, at least the boron or phosphor atoms caught on the bonding interface between the test wafers 1 and 5 diffuse into the regions near the bonding interface of the wafers 1 and 5 and are, thus, conserved.

In step e) of Fig. 1, the bonded wafer pairs 3,6 and 1,5 are separated after annealing with a razor blade 12 which is pushed between the bonded surfaces 4,17 and 2, 16.

Step f) of Fig. 1 shows the wafers in a state after separation wherein at least in a region near the surfaces 2 and 16 of the wafers 1 and 5, the boron or phosphor content is increased due to the diffusion of these particles into the silicon wafers 1,5.

In step g) of Fig.1, the atomic concentration profiles of the wafers 1 and 3 are analysed using a Secondary Ion Mass Spectroscopy (SIMS) device 13. According to a profile 14, measured over about 200 nanometers of the surface beginning from the reference wafer 3, the boron or phosphor concentration at the reference surface 4 of the reference wafer 3 is nearly constant, whereas an atomic concentration profile 15 evaluated at the test surface 2 of the test surface 1 shows especially near the surface an increased concentration of boron or phosphor.

According to step h) of Fig. 1, the atomic concentration profiles 14 and 15 are compared with each other calculating a difference between the profiles. An integration of the profiles between the respective surface and bulk level results in a value for a deposited dose which is commonly expressed in atoms per  $\text{cm}^2$ . The calculated difference is a direct indication of the boron or phosphor concentration of the atmosphere in the clean room because there is

a direct proportional relationship between the boron or phosphor content of the test wafer and the boron or phosphor contamination of the atmosphere in the clean room.

Although the method shown with reference to Fig. 1 is described for silicon wafers, the method can also be applied to any substrates which are able to catch or conserve particles in an atmosphere. Furthermore, the effect of catching and conserving can be attained in a single method step. In the aforementioned described advantageous embodiment, said effect is realised by subsequently bonding and annealing. It is also conceived that said other substrate which is bonded to said test wafer can be a different material than the test wafer.

Further, the wafers can be annealed at any temperature and in any annealing time making it possible that the particles such as boron or phosphor can be conserved by the used substrates. The annealing time and the resulting diffusion depth of the targeted species or particles are dependent on the annealing temperature and the diffusion coefficients of the respective species. The analysed depth  $d$  of the test wafer and of the reference wafer must be higher than the diffusion length of the analysed particles or contaminants. A conventional SIMS-measurement for analysing of the substrates can be replaced by a TOF-SIMS (Time-of-Flight-Secondary Ion Mass Spectroscopy) surface analysis.

Although, the method further described with reference to evaluating the boron or phosphor particle concentration in an atmosphere, the method can further be applied for evaluating any other particle concentration or another type of contaminant in an atmosphere.

Instead of the steps e) and f) of Fig. 1, the caught and conserved particle amount can also be made accessible for analysing by an abrasion of at least one of the wafers 1 or 5 shown in Fig. 1 e) of one of their back sides. By a stepwise backwards abrasion of the material of at least one of these wafers, it can be approached to the region of interest in which particles from surfaces 2,16 can be diffused to analyse this region.

Figure 2 shows schematically a concentration profile of the bonded test wafer pair 1,5 after the annealing step, in which the concentration of diffused contaminants such as boron or phosphor is shown as function of the depth. The depth  $d=0$  corresponds to the respective surfaces 2,16 of the wafers 1,5.

Figure 3 shows schematically a SIMS-concentration profile 21 of the test wafer 1 in comparison to a SIMS-concentration profile 20 of the reference wafer 3, in which the concentration of diffused contaminants is shown as a function of the analysed depth  $d$ . The depth  $d=0$  corresponds to the respective surfaces 2,4 of the wafers 1,3. The near surface concentration of the test wafer 1 of the targeted species like boron or phosphor is higher than the near surface concentration of the reference wafer 3. In order to evaluate the surface dose of contaminants, the difference 22 between the test profile 21 and the reference profile 20 is integrated.

## CLAIMS

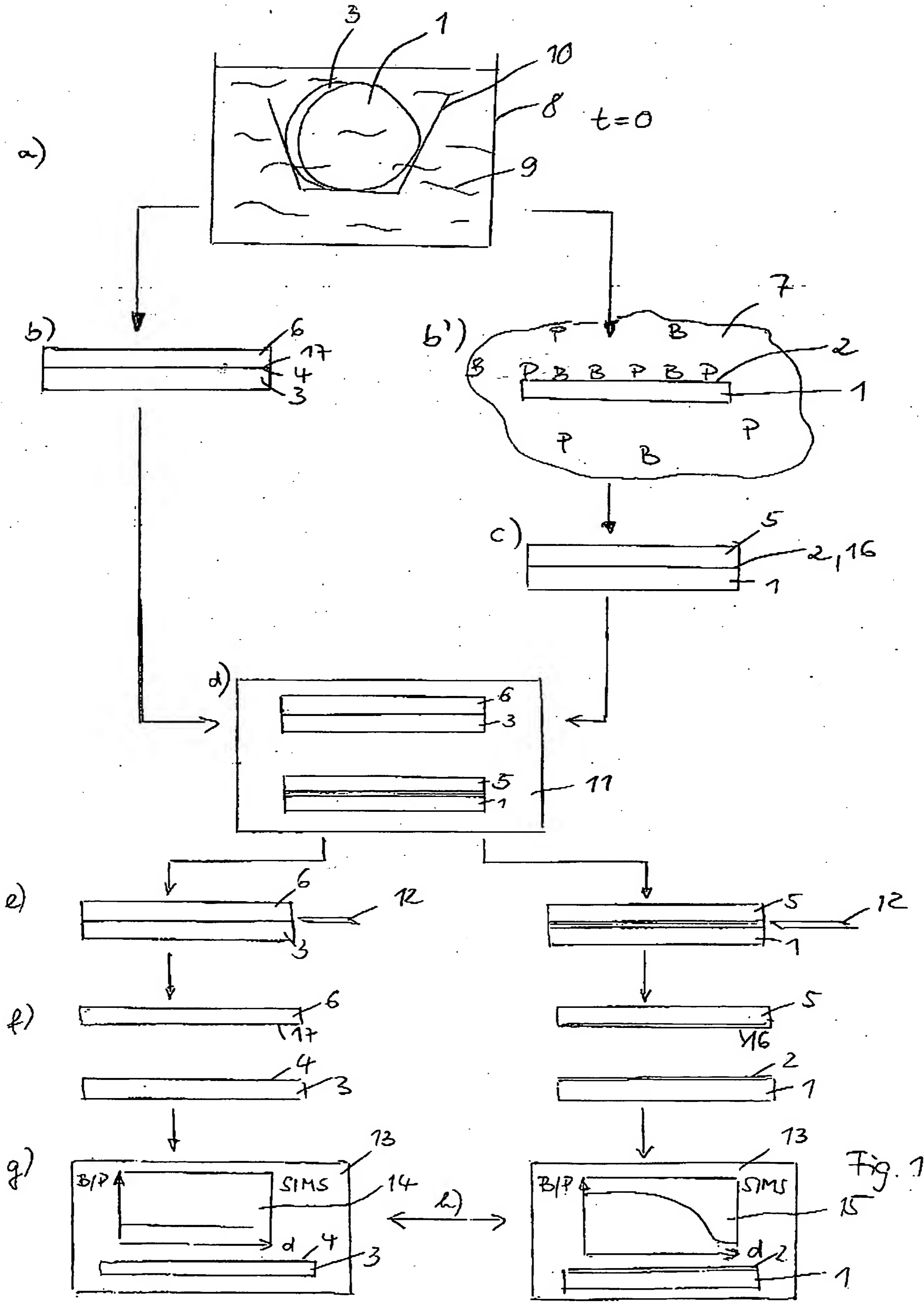
1. A method for evaluating a particle concentration in an atmosphere (7) of a clean room or a machine mini-environment, comprising the steps of  
exposing a test surface (2) of a test substrate (1), especially a silicon wafer, to said atmosphere (7) for a test time (t),  
catching and conserving a particle amount at the test substrate (1) at the end of said test time (t),  
analysing the particle amount and  
comparing the analysed particle amount with a particle amount analysed on a reference substrate (3).
2. A method according to claim 1, **characterised in that** the method further comprises cleaning of the test substrate (1) before exposing the test substrate to said atmosphere (7).
3. The method according to at least one of claims 1 or 2, **characterised in that** the step of catching the particle amount comprises covering the test surface (2) of the test substrate (1) with a surface (16) of another substrate after the test time (t).
4. The method according to claim 3, characterised in that said other substrate is a further test substrate (5), especially a silicon wafer, wherein said substrates (1,5) form a test pair of substrates.
5. The method according to at least one of the preceding claims, **characterised in that** the step of conserving the particle amount comprises an annealing of the test substrate (1) after catching the particle amount.
6. The method according to claim 5, **characterised in that** annealing is performed in a time range between about 1 to about 3 hours, preferably about 2 hours.

7. The method according to at least one of the claims 5 or 6, **characterised in that** said annealing is performed at a temperature between 800°C and 1050°C, preferably at about 950°C.
8. The method according to at least one the preceding claims, **characterised in that** a test pair of substrates (1,5) bonded to each other at the end of said test time (t) is subsequently separated for analysing.
9. The method according to at least one of the preceding claims, **characterised in that** the method further comprises an abrasion of at least one of the back sides of a test pair of substrates (1,5) bonded to each other at the end of the test time (t).
10. The method according to at least one of the preceding claims, **characterised in that** analysing the particle amount comprises evaluating an atomic concentration profile of the test substrate (1).
11. The method according to claim 10, **characterised in that** the atomic concentration profile is evaluated by a Secondary Ion Mass Spectroscopy (SIMS).
12. The method according to at least one of the claims 10 or 11, **characterised in that** the atomic concentration profile is evaluated at the test surface (2) of the test substrate (1).
13. The method according to claim 12, **characterised in that** the test surface (2) is analysed over a thickness (d) of about 100 to 500 nanometers, preferably of about 200 nanometres.
14. The method according to at least one of the preceding claims, **characterised in that** a series of test substrates (1,5) is produced in timely intervals, preferably about every 30 minutes.
15. The method according to at least one of the preceding claims, **characterised in that** the method further comprises cleaning of the reference substrate (3), preferably together with the test substrate (1).

16. The method according to at least one of the preceding claims, **characterised in that** the method further comprises covering a reference surface (4) of the reference substrate (3) with the reference surface (17) of another reference substrate (6).
17. The method according to at least one of the preceding claims, **characterised in that** the method further comprises an annealing of the reference substrate (3).
18. The method according to claim 17, **characterised in that** the annealing temperature and annealing time are equal to an annealing temperature and an annealing time of the test substrate (1).
19. The method according to at least one of the preceding claims, **characterised in that** the method further comprises analysing a particle amount of the reference substrate (3).
20. The method according to claim 19, **characterised in that** analysing the particle amount comprises evaluating an atomic concentration profile of the reference substrate (3).
21. The method according to claim 20, **characterised in that** atomic concentration is evaluated by a Secondary Ion Mass Spectroscopy (SIMS).
22. The method according to at least one of the claims 20 or 21, **characterised in that** the atomic concentration profile is evaluated at a reference surface (4) of the reference substrate (3).
23. The method according to claim 22, **characterised in that** the reference surface (4) is analysed over a thickness (d) of about 100 to 500 nanometers, preferably of about 200 nanometers.

### ABSTRACT

The present invention relates to a method for evaluating a particle concentration, especially of boron or phosphor, in an atmosphere of a clean room or a machine mini-environment. It is the object of the invention to provide such a method of concentration of the clean room or the mini-environment in a short time. The object is solved by a method according to the present invention which comprises the steps of exposing a test surface of a test substrate, especially a silicon wafer, to the atmosphere for a test time; catching and conserving a particle amount at the test substrate at the end of said test time; analysing the particle amount; and comparing the analysed particle amount with a particle amount analysed on a reference substrate.



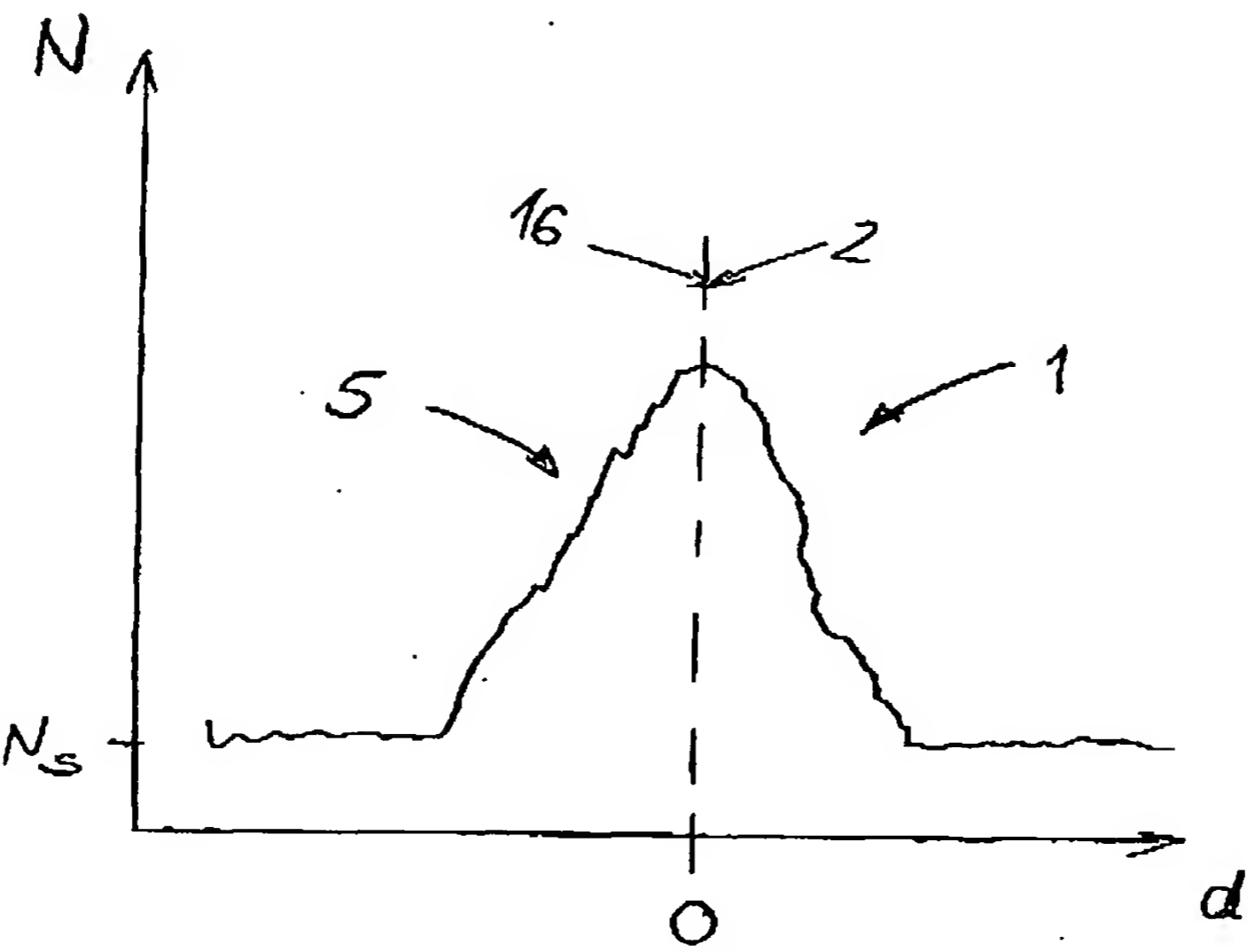


Fig. 2

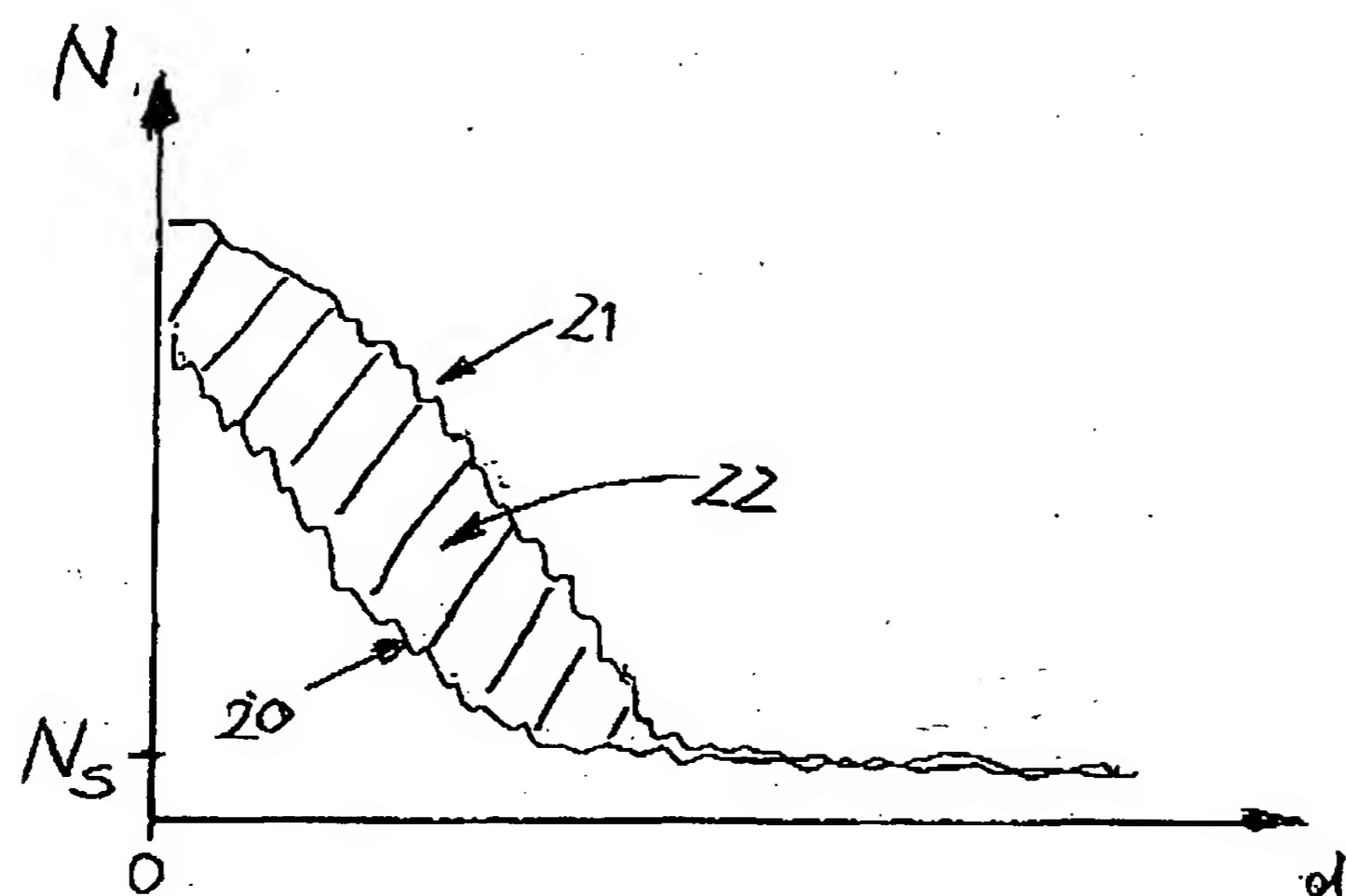


Fig. 3

